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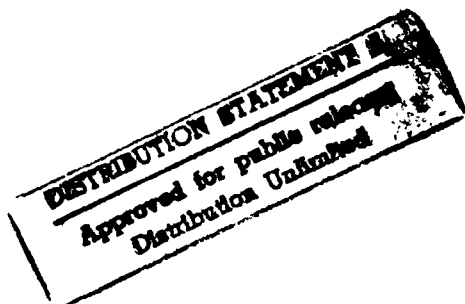
IMPROVED PROCESS FOR FABRICATION
OF COMPOSITE FLEXTENSIONAL SHELLS

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ABSTRACT

Manufacturing of thick walled composite flextensional shells by filament winding has proven to be the most economical manner to make them. However, problems arose with the shells delaminating during production or application of external loads. It has been identified that a major source of the delamination problem is insufficient interlaminar shear strength in the glass/epoxy laminate. Improvements in the order of 30% were achieved, as measured by apparent short beam shear. This was consistently accomplished by minimizing entrained air, controlling winding tensions which produced the correct fiber volume, and using resin additives which assisted the bonding of the epoxy resin to the glass fiber.

TABLE OF CONTENTS

	<u>page</u>
1.0 Introduction	1
2.0 Scope of Work	
2.1 Reduction of Void Content	2
2.2 Improve Fiber/Resin Ratio	2
2.3 Substitute Resin Systems	3
3.0 Discussion	
3.1 Reduction of Voids	4
3.2 Fiber/Resin Ratio	
3.3 Substitute Resin Systems	
4.0 Conclusions/Recommendations	
5.0 References	

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1.0 INTRODUCTION

The manufacturing of thick walled composite flextensional shells by filament winding has proven to be the most economical manner to make them. However, problems with the shells during manufacturing have tended to lower the laminate properties below design requirements. These problems are combinations of shell geometry, fiber wind angle, basic resin properties, and process variables such as tension and fiber content. This study is an attempt to find a solution to allow successful shells to be consistently manufactured. The basic shape of the structure to be wound is shown in Figure 1.

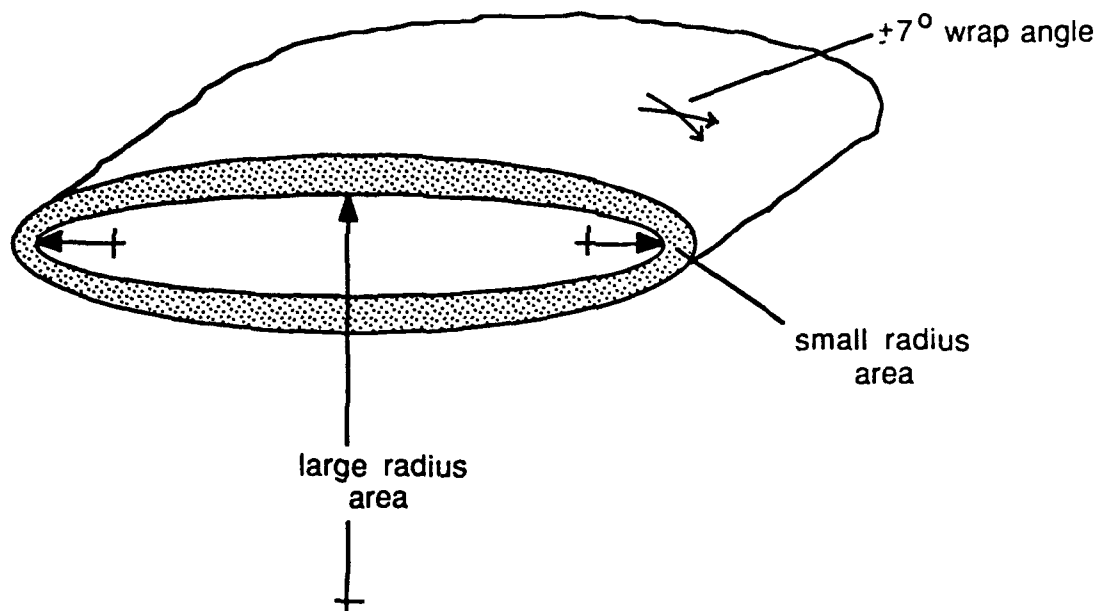


Figure 1. sketch of typical flextensional shell

It has been identified that a major source of the delamination problem, both in manufacturing and in use, is insufficient interlaminar shear strength in the glass/epoxy laminate. This report documents the work that was done in an attempt to control this problem by normal filament winding methods.

2.0 SCOPE OF WORK

The study begins an attempt to bring to acceptable levels in thick parts those parameters which in thinner composites mean high quality laminates. These typically are void contents less than 2% and fiber volumes of $60 \pm 5\%$. Proper resin selection for the application is also important.

2.1 REDUCTION OF VOID CONTENT

The interlaminar shear strength of a composite is a matrix dominated property. It is very dependent upon imperfections in the matrix, the interfacial properties between the fiber and the matrix, and the strength of the matrix itself. Inclusions or voids in the matrix results in internal stress concentrations which increase the chances of cracking and/or failure. If these voids should be located at the interface of the fiber with the matrix, the same results will occur. Therefore, various methods were pursued for minimizing voids resulting from entrained volatiles.

- a) reduction of winding speed
- b) elimination/selective reuse of resin
- c) use of multiple baths/novel impregnation techniques
- d) deaeration of resin
- e) use of antifoaming agents
- f) use of wetting agents

2.2 IMPROVE FIBER/RESIN RATIO

For a given fiber/resin system there is an optimum fiber volume percent ratio that will produce the maximum shear strength for that system. If the percent fiber by volume is increased above this optimum value, the ability of the laminate to transfer loads, from fiber to fiber, through a shear deformation is decreased - there is less resin to transfer the load. Also, with less resin the inclusions play a greater role as stress risers. On the other hand, if the fiber volume is lower than the optimum, the weaker resin begins to carry the tensile loads and fails sooner. Experience has shown optimum fiber ratio to be 58-62% by volume. To this end, attempts were made to reduce the excessive fiber volume caused by high

compaction in the smaller radius areas and to optimize the total part fiber volume. The approach was by the:

- a) controlling of overall fiber tension
- b) introduction of inserts in the small radius area
- c) equalization of compaction in the small vs large radius areas

2.3 SUBSTITUTE RESIN SYSTEMS

A more radical approach to increasing the interlaminar shear strength of the laminate may be a replacement resin which would be amenable to the winding process but would improve the shear strength. Also available are resin additives which can be used to tailor the system to the design requirements. Investigations of candidates to accomplish the above has begun in two areas:

- a) additives to be used in current resin
- b) filament winding resins with improved shear strength

Testing and evaluation are planned on a subscale model.

3.0 DISCUSSION

The source of voids that was minimized was that of air captured in the resin during mixing of the resin and in application of resin on the fiber. Fiber volume was controlled by the amount of tension applied to the fibers. The resin was verified to be the proper system for producing a strong, flexible matrix.

3.1 REDUCTION OF VOIDS

One source of the voids seen in the fabrication of these glass/epoxy flextensional shells is the result of entrained air in the resin. Initially the problem was thought to be due entirely to the foaming created in the resin bath. As the dry fiber entered the bath it brings with it trapped air. It was thought that by slowing down the winding speed, the quantity of air trapped in the resin would be reduced. This approach did not eliminate the real problem, only minimized it. We also did not

fully understand the total effect of this variable (winding speed) on some of the other processing variables (resin application, tension control, fiber volume). These interactions are discussed later on. For these reasons the winding speed was held constant throughout all the studies.

What was done to minimize entrained air was to selectively re-use clear resin during processing of a particular part. Normally, excess resin that drips off any part being wound is collected in a trough and put back into the resin bath. The penalty for eliminating this practice is the increase in cost of the resin and of preparing and mixing it. The benefit is a reduction of voids caused by resin with entrained air. In addition, any broken fibers that may have dropped off are not put back into the resin system. However the total problem of aeration is still not addressed. Air is still captured in the resin from mixing and fiber passing through the bath. Further developments made in these areas are discussed later in this section. The use of an antifoaming agent in the resin, the changes made in the resin application, and the screening of collected resin has enabled the selective re-use of resin to be a viable approach. More minor improvements are being made as studies progress.

The area of resin application is one of the crucial steps in successful filament winding. With proper fiber wet-out (the application of the resin to the fiber) the composite part can achieve its optimum properties for the given fiber/matrix system. Basically, to achieve this, the dry fibers must be spread out and the resin must fully coat each fiber. Any air entrained in the resin will attach itself to the fiber and prevent complete fiber wet-out. The discussion here is limited to ways of reducing voids caused by entrained air during resin application.

In the past, resin was applied using the dip impregnation bath shown schematically in Figure 2a. Here, a bundle of dry fiber is submerged in a bath of resin. As the bundle of fibers plunges into the resin it draws with it any air trapped between fibers. It will also pull in any air next to the surface. Then as the bundle passes through the resin, a turbulent flow is generated within the bath. The flow also draws in some amount of air. As it is pulled out the other side, a squeegee

blade (doctor blade) scrapes excess resin from the fibers in an attempt to control the amount of resin going onto the part. At one point a double bath application was tried to determine whether this set-up would minimize aeration of the resin. However, this procedure was not successful even with the use of a deaerator in the resin.

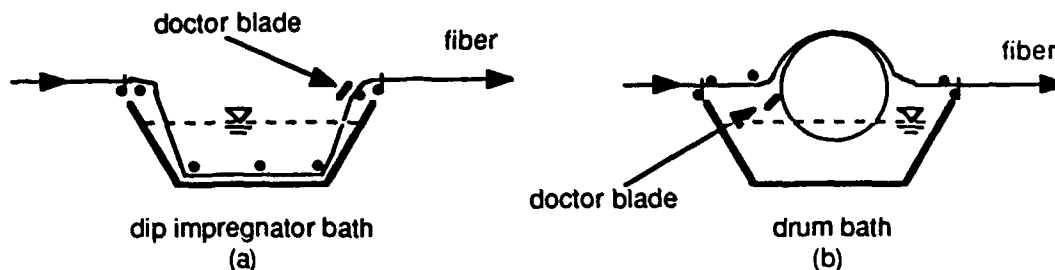


Figure 2. fiber impregnation baths

What has been successful at significantly reducing the entrained air in the resin is the drum bath shown schematically in Figure 2b. The dry fiber bundle is drawn over a smooth drum surface which has been coated with a prescribed amount of resin. Resin is forced through the fiber bundle by the compaction pressure of this fiber on the drum surface. The flow of resin pushes any air to the outside of the bundle as the fibers are completely coated. Mixing of air into the resin is virtually eliminated at this stage due to the smooth motions of the drum and the squeegeeing of resin prior to application on the fibers.

For resin application, the prototype drum bath used in these studies will undergo further refinements but its basic characteristics will remain unchanged.

As mentioned earlier, aeration of the resin can also occur in the preparation and mixing of the resin. The high shear required for blending of the components draws air in. Several solutions have been offered. Mixing could be done in a vacuum, or the blended resin could be deaerated prior to and during fabrication. Some preliminary efforts at degassing during fabrication indicate that although these solutions appear straightforward, their application involves some complications.

A 500 ml sample of resin was taken during fabrication. Vacuum degassing of the sample caused it to froth up to a

volume of 1500 ml. For these shells resin is mixed in 5 gallon batches. It is expected that the increase in volume under vacuum is not linear and would present real problems. Other commercial deaerating methods are being investigated. Centrifugal spinning of quantities of resin is not suggested as it tends to separate the blended components.

The function of an antifoaming agent in a resin is to coalesce small entrained air bubbles into larger bubbles which have a greater buoyancy. Some of the older shells made with the dip impregnation bath had an antifoam agent (ByK®-A501, ByK-Chemie) added to the resin. The foaming problem in the bath was significantly reduced but still present. When the resin application was switched to the drum bath things were further improved. The air bubbles that were present in the bath could be seen percolating to the surface. This antifoaming agent enabled the re-use of resin collected in a drip trough. For all the shells produced with this agent a concentration of 0.2-0.3 parts per hundred parts of resin (phr) was used as suggested by the supplier. Studies will be conducted to determine the optimum concentration for this particular fiber/resin system.

What may be of further assistance in removing entrained air is the addition of a wetting agent to the resin. Typically these agents are to aid in proper and complete fiber wet-out. Their action of reducing surface tension will also help in allowing entrained air bubbles to escape. When this type of resin additive is investigated in future efforts to improve fiber wet out, its effect on entrained air will also be explored.

3.2 FIBER/RESIN RATIO

In filament winding the percent fiber content by volume (FV) is largely dictated by the amount of resin applied to the fiber and the compaction of the fiber onto the tool. Compaction is a function of winding tension in the fiber and tool geometry. For optimum interlaminar shear properties the resin manufacturer recommends a FV between 58% and 62%. Initially we were observing FV of 65% to 77% in the small radius and 62% to 73% in the large radius ends. The application of resin with the dip impregnation bath discussed in the previous section did not allow for much control over the amount of resin applied and the tension set in the fiber.

What has been learned is that it is essential to isolate the resin application from the tension control. This was accomplished with the drum bath. The tension control mechanism that was successfully used throughout the rest of these studies is shown in Figure 3. By way of changing the relative position of four roller wheels, a fixed resistance was put in each fiber. With this mechanism, better FV control was achieved. The tension in the fiber was measured with a spring scale to be around 1.0 to 1.5 lb_f per fiber end. Measurements were made by tying the ends of the fibers to the scale and then drawing the fibers through the production set-up.

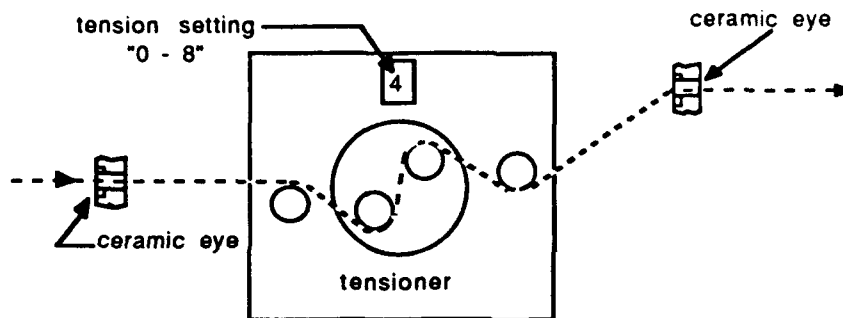


Figure 3. Sketch of tensioner used for center pull fiber packages

As for tool geometry, what has been learned here is that no matter how similar tools may appear, each change in geometry must be treated as a separate case. In general though, as the fiber wraps from the large to the small radius area the normal compaction force increases. The increased force will squeeze more resin out of this end of the part. To solve the problem it was suggested that inserts be used in the small radius ends to either retain or replace the resin being displaced. It should be cautioned here that the full extent of the nature of the cracking problem has not been analyzed. It is possible that transverse tensile stresses may also be contributing to this problem and improper use of inserts could aggravate this problem.

A 0.008 inch thick fiberglass cloth, 1581-I617 (Burlington Glass Fabrics), was used as the insert. The number of inserts used is proprietary to the part, but was based on design requirements.

These were thoroughly wet-out and squeegeed to remove any air bubbles. They were selectively placed in the inner and outer regions of the small radius ends in a way that avoided the maximum shear zone. The result was a typical FV distribution as shown in Figure 4b. Cross-sectional views under the microscope did not show any resin rich areas on the underside of the insert. Shells of this nature were manufactured with no cracking problems.

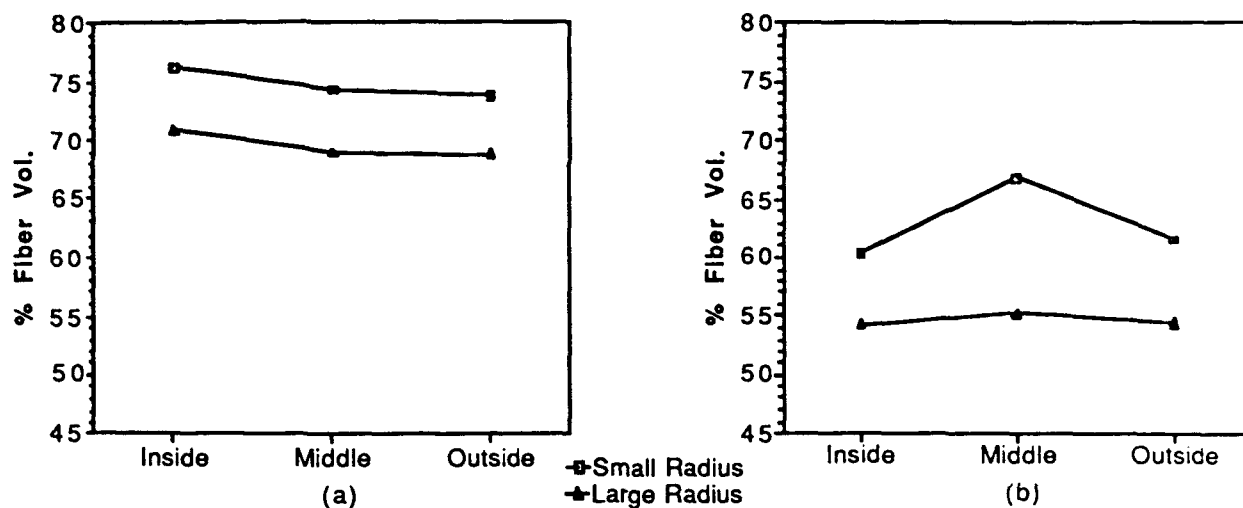


Figure 4. Typical through the thickness fiber volume distribution for flextensional shells; (a) no inserts, (b) 0.008" thick fiberglass cloth inserts

3.3 SUBSTITUTE RESIN SYSTEM

With all other winding variables at optimum conditions, the strength of the matrix itself will effect the interlaminar shear strength of the composite. Studies by Brelant and Petker¹ found a correlation between neat resin tensile strength and interlaminar shear strength. Figure 5 is a graphical representation of their findings. As resin strength is increased an upper limit on interlaminar shear strength is reached. Further increases in tensile strength have little or no effect on

shear strength. The apparent limiting factor is the fiber/matrix bond strength. Further increases in shear strength could only be possible with increases in bond strength.

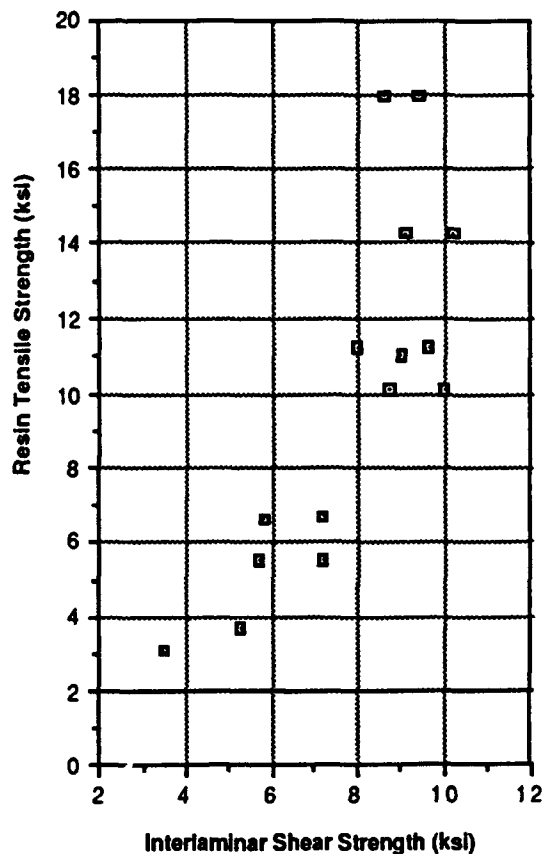


Figure 5. Data from Brelant and Petker¹

Table 1 shows a variety of typical filament winding epoxy resins and their tensile properties. The Shell EPON® 9405/9470 (100:37 mix ratio) was used on these shells because of its long pot life and high strength. When looking at the bond strength of this high strength resin to the fiber the first concern is to make sure the proper, and best, sizing agent is used on the fiber. A sizing agent is a coating applied to the fiber to enable the matrix to bond to it. In this case, with glass fibers and epoxy resin, a silane type sizing is recommended and being used. Thorough understanding of these sizings is hard to gather because they are proprietary to each glass manufacturer. Owens-Corning 346-BA450 glass, currently

being used, is a silane sized fiber. Other fiberglass manufacturers and sizing types will be investigated with this resin system and any other resins which may be investigated.

Table 1. Typical filament winding resins

RESIN SYSTEM	POT LIFE (hours)	VISCOSITY (cps)	TENSILE STR. (ksi)	TENSILE MOD. (ksi)	ULTIMATE STRAIN %
SHELL					
9405/9470 *	54	950	11.3	413	6.4
9405/9470 **	41	850	11.7	427	8.5
DPL 9420/9470 *	20	900	8.3	386	3.1
DPL 9420/9470 **	20	900	11.2	411	5.2
9102/9150	8	800	11.0	520	3.5
DOW TACTIX					
123/H41	6	500	11.1	430	5.5
DOW DER EPOXY					
331/Anhydride	6	1000	12.1	400	n/a
338/Anhydride	8	740	11.5	400	n/a

* Mix ratio of 100:28

** Mix ratio of 100:37

Even with the best combination of fiber, sizing, and epoxy resin, deleterious stress concentrations do occur at the interface. These are due to the mismatches in the mechanical stiffness of the fiber and the matrix. The ideal interface would be successive layers of varying modulus around the fiber that would more efficiently transfer the loads to the matrix. The addition of elastomers and "fortifiers" to the resin have been tried by others with some success. These studies are currently being investigated.

What has shown a significant improvement on the interlaminar shear strength was the addition of a coupling agent to the resin. The coupling agent acts as a molecular bridge, rather than a coating, at the fiber/matrix interface. A neoalkoxy zirconate coupling agent (LZ97, Kenrich Petrochemicals) added to the resin did help increase the apparent short beam shear. It's exact contribution to the increase is not clear, because curing parameters were also changed, but the total increase was in the

order of 30%! This parameter will be isolated to determine its full value as well as the optimum mix concentration.

4.0 CONCLUSIONS/RECOMMENDATIONS

Further investigation could be done to verify the ideal antifoam and wetting agents, and their respective concentrations, for use with the EPON® 9405/9470. Other resin systems or other fibers may yield some increases in interlaminar shear strength. A feasible method of deaerating the resin during mixing and in-process may be found. But still not understood is the state of transverse stress and the effect of fiber tension on part performance.

The overall result of these studies has been a significant improvement in the manufacture of flextensional shells as well as an appreciation for further refinements that may lead to improved performance. The major factors contributing to the elimination of cracking in the small radius corners was the reduction of voids, the use of a coupling agent, and the separation of the resin application from the tension control. Changes in the cure cycle were also a major contributing factor².

Until other developments are made, it is recommended that the flextensional shells continue to be manufactured with the EPON resin/Byk- A501/LZ 97 resin mixture. A drum type resin application and a moderate tension setting of approximately 1-1.5 lb_f should be used. This tension should be adjusted to yield a part with a fiber volume of 58% - 62% by volume. These can only be considered general rules because any changes in tool geometry results in changes in processing variables. The use of cloth inserts in the small radius area does seem to help in controlling the fiber volume and part thickness, but its complete effect on part performance should be analyzed.

5.0 REFERENCES

footnotes:

1. S. Brelant and I. Petker, " Fabrication and Environmental Interaction Effects of Filament-Wound Composites", Mechanics of Composite Materials, (Eds. F. W. Wendt, H. Liebowitz and N. Perrone), Pergamon Press, Oxford, 1970.
2. M. E. Deckers, "Minimization of Thermal Stresses in Filament Wound Flextensional Shells", Naval Research Laboratory - USRD, Florida, Contract No. N62190-88-M-1731, 1988.

filament winding diagram:

